INDUSTRY CIRCULAR

OFFICE OF THE COMMISSIONER OF INTERNAL REVENUE
ALCOHOL AND TOBACCO TAX DIVISION

WASHINGTON 25, D. C.

Industry Circular No. 59-47

July 8, 1959

CARBON DIOXIDE TEST PROCEDURES

Proprietors of bonded wine cellars
and others concerned:

Purpose. The purpose of this industry circular is to announce authorized test procedures for determining carbon dioxide in still wines where such carbon dioxide is added to or retained in the wines under the provisions of 26 CFR 240.531 through 240.534.

Background. Public Law 85-859 amended the Internal Revenue Code of 1954 to provide that limited quantities of carbon dioxide may be added to or retained in still wines. These provisions were implemented by amendments of 26 CFR Part 240. Such amendments provided that authorized test procedures for determination of the quantity of carbon dioxide in still wines would be announced by the Director, Alcohol and Tobacco Tax Division.

Procedures authorized. The authorized procedures for determining the quantity of carbon dioxide in still wines are described in Exhibit 1 and apparatus used in the procedures is depicted in Exhibits 2, 3, and 4. Two procedures are provided. One is designated as the "Gasometric Procedure" and the other is designated as the "Titrimetric Procedure." Either procedure may be used. The method of preparation of the sample is common to both procedures.

Inquiries. Inquiries in regard to this industry circular should refer to the number thereof and should be addressed to the office of your assistant regional commissioner (alcohol and tobacco tax).

Dwight E. Avis
Director, Alcohol and Tobacco Tax Division

Attachments:
Exhibits 1, 2, 3, and 4

IRS-D.C.-50287

EXHIBIT 1

AUTHORIZED TEST PROCEDURES

Preparation of samples. Chill the unopened bottle of wine in an ice-salt bath which is slightly below 32° F. (30 minutes for 1/10 and 1 hour for 1/5 gallon bottles). Open the bottle of chilled wine and add 1.5 ml of 50% sodium hydroxide per 100 ml of wine. Stopper the bottle and agitate.

Reagents: (Common to both procedures)

Phosphoric Acid - reagent grade (85%)
Hydrogen Peroxide - 10% (For destruction of sulfur dioxide)
Sodium or potassium bicarbonate - reagent grade
(Before use, dry over sulfuric acid for 24 hours at room temperature)
Sodium Hydroxide - 50% by weight - Dissolve 500 grams of solid sodium hydroxide in 500 ml of water, allow to stand 24 hours and filter before use.

Gasometric procedure.

Standard sodium bicarbonate solutions:

Weigh the following quantities from a freshly opened bottle of dried sodium bicarbonate and transfer to a liter flask with approximately 700 ml of recently boiled and cooled distilled water. Add 200 ml of neutral absolute alcohol, shake, cool and make to mark with boiled distilled water:

3.8182 grams = 200 milligrams CO_2/100 milliliters
4.2955 grams = 225 milligrams CO_2/100 milliliters
4.7727 grams = 250 milligrams CO_2/100 milliliters
5.2500 grams = 275 milligrams CO_2/100 milliliters
5.7273 grams = 300 milligrams CO_2/100 milliliters

Apparatus:

Carbon Dioxide Apparatus - Shown in Exhibits 2 and 3.
Magnetic Stirrer with Teflon Stirring Bar (Fisher Fexa-Mix or equivalent). The stirring bar should be 1 to 1-3/8 inches in length.
Vacuum Pump - Welch Dist-O-Pump, with motor single stage, vented exhaust; to be operated with vented exhaust

Exhibit 1 (continued)

valve open for pumping condensable vapors. Insert a three-way stopcock between the pump and the apparatus for allowing air to enter the system.

Grease - Silicone, high vacuum type. To remove this grease from the glassware, use varsol or hot kerosene.

Calibration:

Pipette 50 ml of standard sodium bicarbonate solution and 3 ml of hydrogen peroxide solution into the reaction flask. Carefully grease the joints then start the magnetic stirrer and evacuate the system for one minute. (Longer periods cause excessive cooling necessitating a waiting period for reestablishment of temperature equilibrium.) Close the system to the pump (by three-way stopcock), gently tap the mercury columns and read the mercury levels of the manometer to the closest 0.5 mm. At this point the mercury levels should remain constant. (Changes indicate a leak, probably caused by insufficient grease on the joints.)

Add 10 ml of phosphoric acid and continue with rapid stirring for five minutes. Gently tap the mercury columns and read the total pressure in cm mercury to the nearest 0.5 mm. Record the room temperature in degrees centigrade.

Open the three-way stopcock (on the apparatus) to the pump. Then, slowly open the three-way stopcock between the pump and the apparatus to permit air to flow into the system. Disconnect and thoroughly wash the inner portion of the acid dispensing unit and reaction flask. Rinse with acetone (technical grade) and dry with suction.

Calculations:

Calibrate with each standard solution in duplicate and calculate the average volume of the system as follows:

From the total pressure in cm mercury, subtract the vapor pressure of the ethanol-water, and the pressure due to the phosphoric acid effect (See Table 1).

Exhibit 1 (continued)

TABIE 1 - Effect of Phosphoric Acid on the Vapor Pressure of Ethanol and Water

Percent Ethanol	Increase in pressure after Phosphoric Acid Added in cm Mercury
Pure water	0.67
5	0.68
10	0.69
15	0.75
20	0.77
25	0.77

Then:

(Equation 1)

$$V = \left[\frac{0.08205 \quad (273 + {}^{o}C) \quad \frac{g}{M} \quad 76}{P} \right]$$

V = The volume of the system in liters.
0.08205 = Gas constant in liter atmosphere per degree per mole.
273 + °C = Absolute temperature plus the room temperature in °centigrade.
g = The weight in grams of carbon dioxide in 50 ml sample.
M = The molecular weight of carbon dioxide in grams.
P = The pressure of carbon dioxide in cm mercury.

Correction for the mercury displaced in the manometer tubing:

(Equation 2)

$$V_m \quad \frac{L}{2} \pi r^2$$

L = Difference in height of mercury column in cm.
r = Radius of manometer tubing.
V_m = Volume correction - In calibrating the volume of the system, subtract V_m from the uncorrected volume, V; then:

Exhibit 1 (continued)

(Equation 3)

V_o (calibrated volume of the system) $= V - V_m$

In calculating the weight of carbon dioxide in the wine, the mercury displaced in the manometer tubing, V_m, is added to the calibrated volume of the system, V_o; then:

(Equation 4)

$$V = V_o + V_m$$

Analysis of Wine:

Pipette 50 ml of alkaline wine (at room temperature) and 3 ml of hydrogen peroxide solution into the reaction flask and proceed as under calibration. Solve for "g" (grams CO_2 per 100 ml wine) in the following equation using V determined from Equation 4:

(Equation 5)

$$g = 14.327 \left[\frac{PV}{273 + {}^{\circ}C} \right]$$

Titrimetric procedure.

Reagents:

Sodium Hydroxide - approximately 0.25 normal - Prepare from 50% sodium hydroxide. Standardize the sodium hydroxide solution against potassium acid phthalate or other primary standard using phenolphthalein-thymolphthalein indicator.

Hydrochloric Acid - approximately 0.25 normal - Standardize against the sodium hydroxide solution using phenolphthalein-thymolphthalein indicator. (The normality of this solution should remain constant for considerable periods of time.)

Barium Chloride - Dissolve 60-65 g. of hydrated barium chloride in a liter of water and neutralize to phenolphthalein.

Exhibit 1 (continued)

Restandardization: Restandardize sodium hydroxide solution daily versus hydrochloric acid, adding 5 ml of barium chloride solution.

Indicator: 1 g. phenolphthalein plus 0.5 g. thymolphthalein in 100 ml of 95% ethyl alcohol.

Apparatus:

500 ml sidearm distilling flask, Exhibit 4. (Rubber stopper and standard distilling flask may be used.)
Glass tubing - 6 mm connecting distilling flask and carbon dioxide traps.
3 Pyrex test tubes (25 x 200 mm)
3 Gas dispersion tubes - 12 mm fritted ends (coarse porosity) and 8 mm stems (Fisher Catalog 11-138)
Vacuum line or filter pump. A trap should be placed in the line.

Procedure:

Set up the apparatus as shown in Exhibit 4 and place the receivers in a beaker of water (below 80° F).

Pipette 20 ml of the standard sodium hydroxide solution into the first and second receivers and 10 ml of sodium hydroxide plus 10 ml of barium chloride solution into the third.

Pipette 50 ml of alkaline wine into the distilling flask and 3 ml of 10% hydrogen peroxide. Add boiling chips (not marble).

Attach the vacuum line to the last carbon dioxide receiver and slowly increase the vacuum until bubbling practically ceases, then open the vacuum line fully. (This will keep the system under partial vacuum so that on heating, the stoppers will not be blown out by a sudden surge of steam or carbon dioxide.)

Add about 5 ml of 85% phosphoric acid to the dropping funnel and carefully admit about 3 ml into the distilling flask. Next agitate the flask gently to mix the acid and sample.

Exhibit 1 (continued)

Heat gently and when carbon dioxide evolution slows down, heat vigorously, after a few ml of liquid have been distilled and the top of the first receiver is warm, all of the carbon dioxide will have been driven into the receivers.

Close the vacuum line between trap and receivers, then slowly admit air through the dropping funnel until atmospheric equilibrium is reached.

Transfer the contents and rinsings of the first two receivers and spargers into a titration flask, third also if $BaCO_3$ has precipitated.

Add 50 ml of barium chloride solution to the titration flask and titrate with hydrochloric acid to phenolphthalein end point.

Calculations:

(Ml. NaOH x normality) - (Ml. HCl x normality) = ml of normal base consumed by CO_2

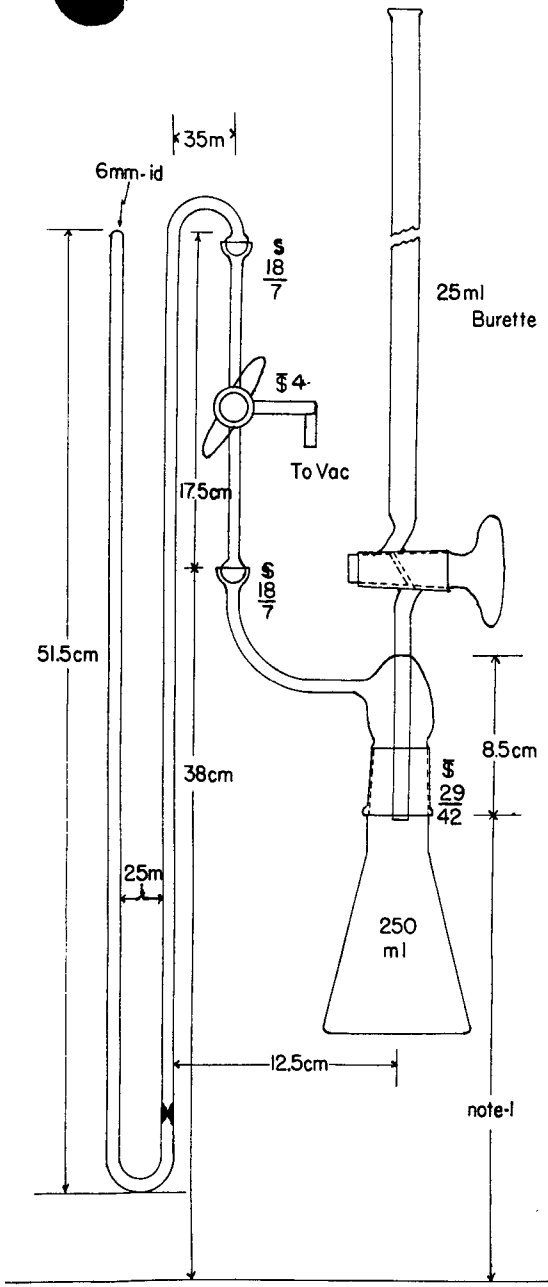
(1 ml of normal $CO_2 \equiv 0.022$ g.)

0.022 x ml NaOH (consumed) x 2 x 1.015 = wt. CO_2 in g/100 ml.

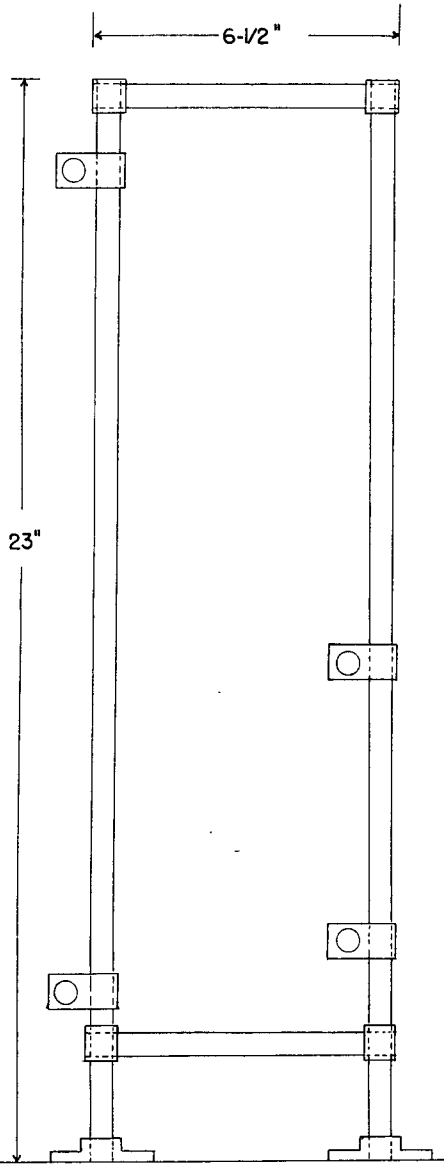
Note: Simulated samples for carbon dioxide determinations may be prepared from sodium or potassium bicarbonate.

Duplicate determinations for carbon dioxide should be made on the wine samples.

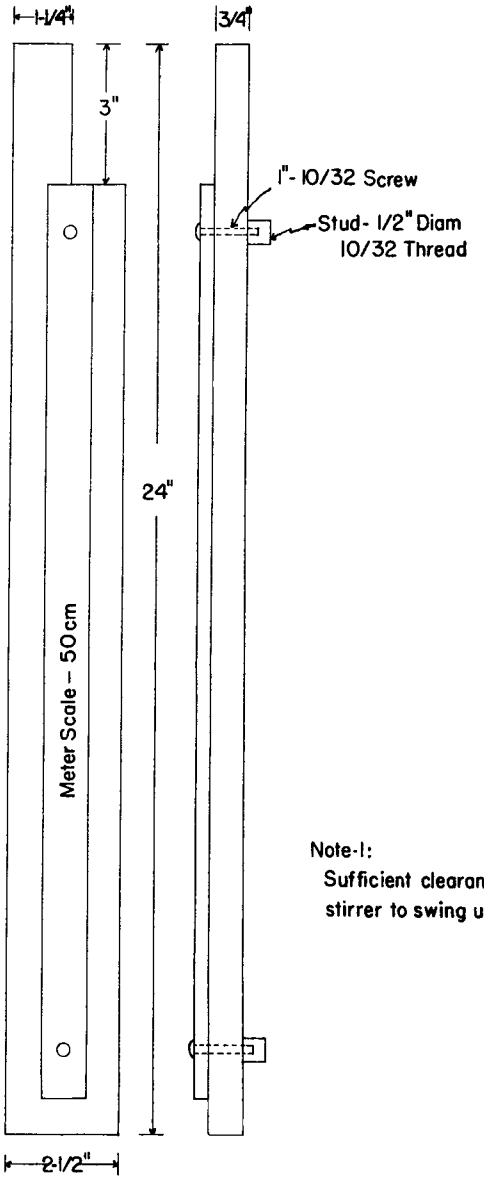
CARBON DIOXIDE DETERMINATION APPARATUS



Manometer-Reaction Flask Assembly



Flexaframe Assembly



Plan View Side View

Manometer Mount

Note-1:
Sufficient clearance for magnetic stirrer to swing under flask

EXHIBIT 2

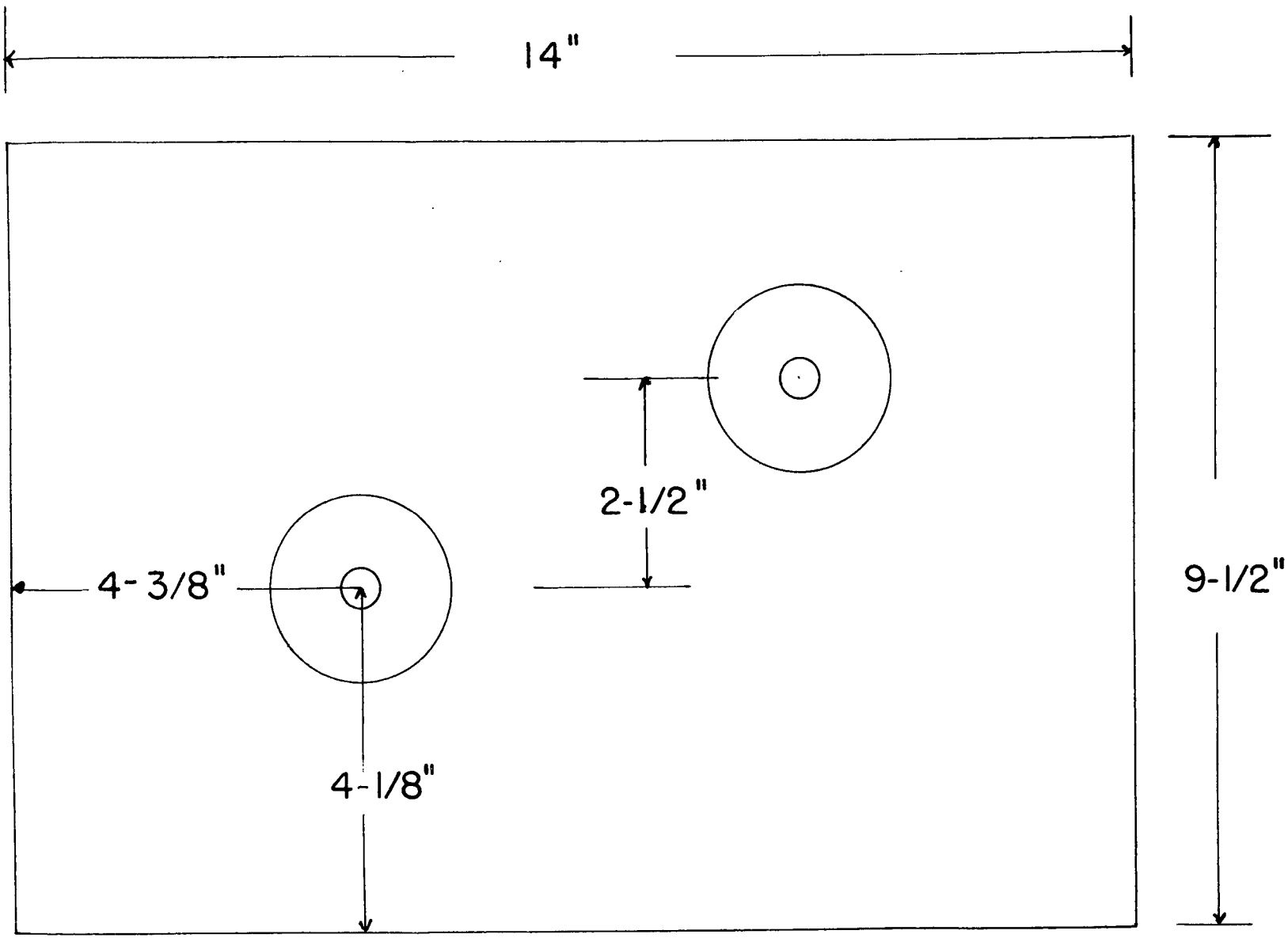


EXHIBIT 3

Base & Foot position

Material: Hard wood - 1" t[illegible]k.

TITRIMETRIC APPARATUS FOR CARBON DIOXIDE DETERMINATION

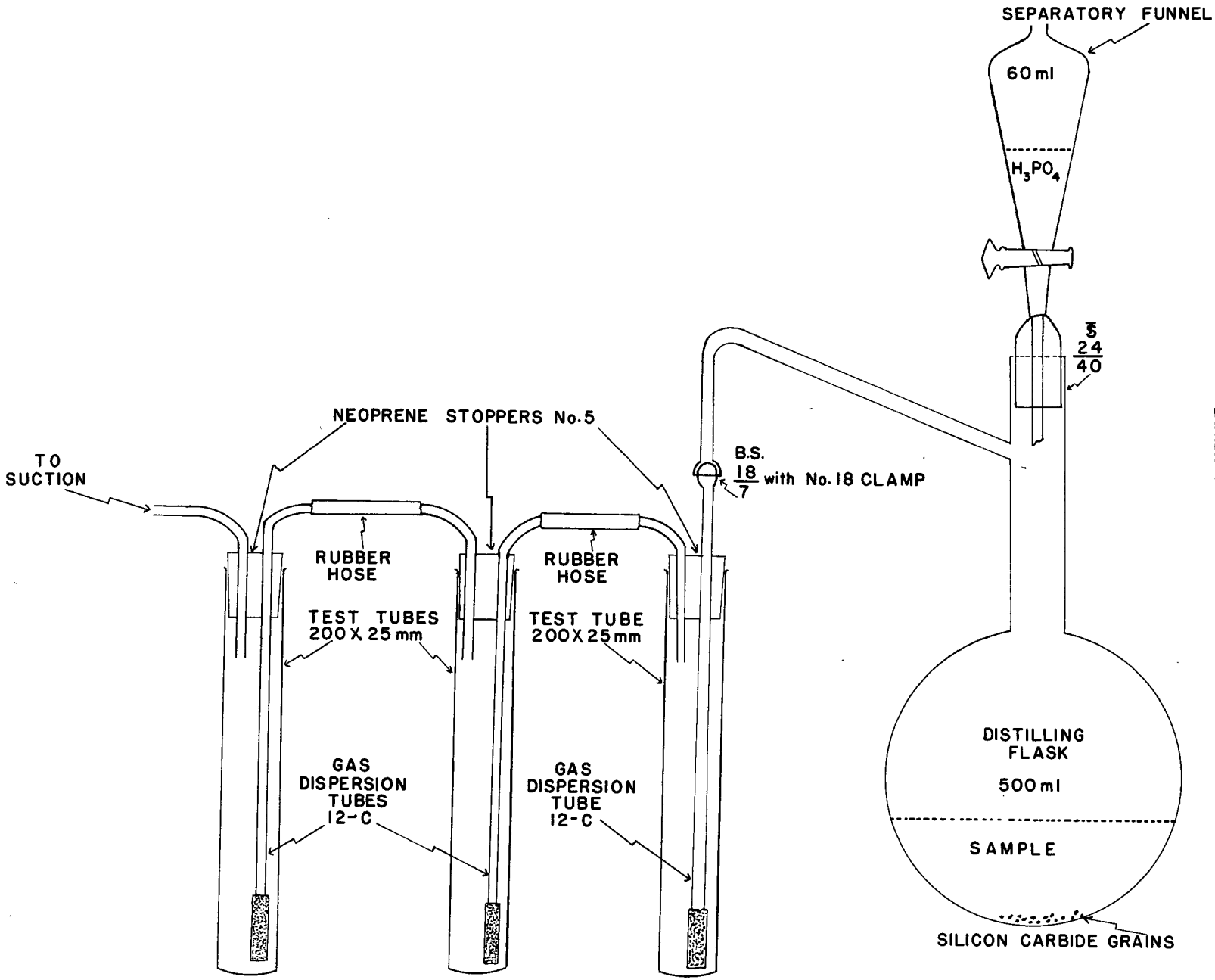


EXHIBIT 4